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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : C11D 17/00, 3/30, 3/26, 3/18, 1/37, 3/065 // 3/39, 3/37, 3/386, 1/62, 3/48, 3/42, 1:14, 1:22	A1	(11) International Publication Number: WO 00/17311 (43) International Publication Date: 30 March 2000 (30.03.00)
(21) International Application Number: PCT/US98/19917		(81) Designated States: AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK (Utility model), SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
(22) International Filing Date: 23 September 1998 (23.09.98)		
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(54) Title: ENCAPSULATED MATERIALS AND BAR COMPOSITIONS CONTAINING SUCH MATERIALS

(57) Abstract

The present invention is directed to an encapsulated detergent particle containing a detergent active encapsulated with a coating material enabling a delayed release of the detergent active into the wash solution, the coating material being insoluble in a wash solution having a pH of equal to or greater than 10 at 25 °C and such coating material being soluble in a wash solution having a pH of equal to or less than 9 at 25 °C. The present invention also relates to detergent bar compositions containing the encapsulated detergent particle.

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ENCAPSULATED MATERIALS AND
BAR COMPOSITIONS CONTAINING SUCH MATERIALS

5

FIELD

10 This invention relates to encapsulated detergent actives and detergent bar compositions comprising such encapsulated materials.

BACKGROUND

15 Detergent compositions contain various detergent actives to improve the effectiveness of cleaning. Common detergent actives that are used in formulating detergent compositions to enhance cleaning performance include bleaches, bleach activators, enzymes, etc.

20 The performance activity of such detergent actives may be limited due to their relative instability in the detergent matrix environment. For example bleaches, especially peroxygen bleaches, decompose rapidly when exposed to moisture. In addition, the detergent actives may also degrade due to process conditions, such as exposure to mechanical shear or high temperature conditions when making the detergent composition. Furthermore, during prolonged storage of the final detergent composition, the detergent active may degrade, thereby providing less than optimum performance when the consumer finally utilizes the 25 detergent compositions.

30 There are various means known in the art to coat or encapsulate detergent actives to solve the problems of instability. One example is to encapsulate the detergent active with a poorly water soluble coating and/or encapsulate with a thick coating. Examples of such coatings include waxes, fatty acids, and celluloses.

35 The performance activity of detergent actives may also be enhanced by delaying the release of the detergent active into the wash solution. Means for altering the physical characteristics of the detergent active to control its solubility and rate of release is known, such as coating with poorly water soluble materials. In addition the wash solution environment in which the detergent active will be

released may be altered, such as high temperature or a change in pH. In one example of a controlled pH release washing method, a chlorine bleach is encapsulated with a coating material which is insoluble in water at pH 7. As the encapsulated bleach is exposed to an aqueous alkaline solution, such as an initial wash solution in which a detergent composition is placed in the wash water, the encapsulated coating swells or dissolves, thereby releasing the bleach.

There is a continuing need to provide means to provide stability of detergent actives. In addition, there is a continuing need to provide means to delay the release of the detergent active into the wash solution.

Applicants have now unexpectedly found that a particular coating material may be used to encapsulate detergent actives so that not only is the stability of the detergent active protected, but the release of the detergent active into the wash solution can be timed to be released to provide the best cleaning performance benefit.

None of the existing art provides all of the advantages and benefits of the present invention.

SUMMARY

The present invention is directed to an encapsulated detergent particle containing a detergent active encapsulated with a coating material enabling a delayed release of the detergent active into the wash solution, the coating material being insoluble in a wash solution having a pH of equal to or greater than 10 at 25°C and such coating material being soluble in a wash solution having a pH of equal to or less than 9 at 25°C. The present invention also relates to detergent bar compositions containing the encapsulated detergent particle.

These and other features, aspects, and advantages of the present invention will become evident to those skilled in the art from a reading of the present disclosure.

DETAILED DESCRIPTION

While this specification concludes with claims distinctly pointing out and particularly claiming that which is regarded as the invention, it is believed that the invention can be better understood through a careful reading of the following detailed description of the invention. In this specification, all percentages, ratios, and proportions are by weight, all temperatures are expressed in degrees Celsius, molecular weights are in weight average, and the decimal is represented

by the point (.), unless otherwise indicated. All documents cited are incorporated herein by reference. Citation of any reference is not an admission regarding any determination as to its availability as prior art to the claimed invention.

As used herein, "comprising" means that other steps and other ingredients which do not affect the end result can be added. This term encompasses the terms "consisting of" and "consisting essentially of".

As used herein, the term "alkyl" means a hydrocarbyl moiety which is straight or branched, saturated or unsaturated. Unless otherwise specified, alkyl moieties are preferably saturated or unsaturated with double bonds, preferably with one or two double bonds. Included in the term "alkyl" is the alkyl portion of acyl groups.

The term "coconut oil" is used herein in connection with materials with fatty acid mixtures which typically are linear and have an approximate carbon chain length distribution of about 8% C₈, 7% C₁₀, 48% C₁₂, 17% C₁₄, 9% C₁₆, 15% C₁₈, 7% oleic, and 2% linoleic (the first six fatty acids listed being saturated). Other sources having similar carbon chain length distribution in their fatty acids, such as palm kernel oil and babassu oil, are included within the term coconut oil.

The term "tallow" is used herein in connection with materials with fatty acid mixtures which are typically linear and have an approximate carbon chain length distribution of 2% C₁₄, 29% C₁₆, 23% C₁₈, 2% palmitoleic, 41% oleic, and 3% linoleic (the first three fatty acids listed are saturated). Other mixtures with similar distribution, such as those from palm oil and those derived from various animal tallow and lard, are also included within the term "tallow." The tallow can also be hardened (i.e., hydrogenated) to convert part or all of the unsaturated fatty acid moieties to saturated fatty acid moieties.

The term "soluble" and "insoluble" is used herein to mean sufficiently soluble to be able to analyze for dissolved material by standard chemical methods.

pKa is used herein in the same manner as is commonly known to people skilled in the art of chemistry. Values referenced herein can be obtained from literature, such as from "Critical Stability Constants: Volume 2, Amines" by Smith and Martel, Plenum Press, NY and London, 1975. Additional information on pKa's can be obtained from relevant company literature, such as information supplied by Dupont. The pKa is an equilibrium constant which can change with temperature and ionic strength; thus, values reported in the literature are

sometimes not in agreement depending on the measurement method and conditions. To eliminate ambiguity, the relevant conditions and/or references used for pKa's of this invention are as defined herein or in "Critical Stability Constants: Volume 2, Amines". One typical method of measurement is the 5 potentiometric titration of the acid with sodium hydroxide and determination of the pKa by suitable methods as described and referenced in "The Chemist's Ready Reference Handbook" by Shugar and Dean, McGraw Hill, NY, 1990.

The present invention is directed to an encapsulated detergent particle containing a detergent active encapsulated with a coating material enabling a 10 delayed release of the detergent active into the wash solution, the coating material being insoluble in a wash solution having a pH of equal to or greater than 10 at 25°C and such coating material being soluble in a wash solution having a pH of equal to or less than 9 at 25°C.

When the encapsulated detergent particle is used in the beginning of the 15 wash, the wash water has a high pH (pH of 10 or greater) due to the addition of the detergent composition into the wash water. Thus, the detergent active remains encapsulated since the coating is not soluble at a pH of 10 or greater.

As the fabric is laundered through the wash cycle, the pH of the wash water lowers over time as the detergent composition becomes more and more 20 diluted, and especially during the rinse cycle when fresh rinse water is added. As the pH of the wash water drops, the coating becomes soluble, thereby releasing the detergent active into the wash. By delaying the solubility of the coating material, one can control the release of the detergent active to a later 25 stage in the wash cycle where the detergent active can provide the most performance benefit. For example, if the detergent active is a polymeric dispersant, better anti-redeposition of soils is achieved if the polymeric dispersant can be released later in the wash cycle, e.g. the rinse cycle, in which redeposition most commonly occurs.

The encapsulated detergent active is protected from the detergent matrix 30 environment that may degrade the detergent active upon exposure. The detergent active is protected from for example, water destabilization, and/or the detergent active has greater resistance to shear forces and temperature changes, and/or is easier to manufacture. An even further additional benefit is that the coating material may protect the detergent active from degradation in a 35 final detergent composition during storage until use by the consumer, as well as

have elastic properties which protects the active ingredient, e.g. cracking from shear forces or extreme temperatures.

An encapsulated particle having an elastic coating layer which deforms, but does not crack when subjected to shear forces, is particularly useful for 5 addition to a laundry bar composition. A typical laundry bar production process subjects the laundry bar composition to shear forces as the composition is mechanically worked. These processing conditions tend to destabilize an active ingredient such as a bleach or an enzyme, unless special process steps are taken. These special process steps are typically expensive, in terms of capital 10 outlays for machinery, slower throughput, etc. However, an elastic coating layer can protect an active ingredient, even from typical laundry bar processing conditions. Thus, the current invention allows an active ingredient to be incorporated into a laundry bar composition, without having to resort to expensive processing changes.

15 **A. Detergent active**

The encapsulated detergent particle of the present invention contains a detergent active which has an activity when used in a cleaning composition. The detergent active can be any known commonly used detergent actives used in detergent bar compositions in which it would be beneficial to protect the stability 20 and/or perform more effectively if released later in the wash cycle. Preferred detergent actives include oxygen bleaches, bleach activators, polymeric soil release agents, enzymes, fabric softening agents, bactericides, bacteriostats, suds suppressors, and mixtures thereof.

Preferably the detergent actives are solids and are not liquids or 25 coacervates.

The preferred detergent actives are described in detail below. It is also preferred to have these detergent actives as ingredients in detergent compositions containing the encapsulated detergent particle of the present invention, independent of the fact that the same detergent active or combinations 30 of the detergent actives are used in the encapsulated detergent particle.

1. Oxygen bleach

The encapsulated detergent particle of the present invention may contain an oxygen bleach. Oxygen bleaching agents useful in the present invention can be any of the oxidizing agents known for laundry, and other cleaning purposes, 35 including hard surface cleaning. Oxygen bleaches or mixtures thereof are

preferred, though other oxidant bleaches, such as oxygen, an enzymatic hydrogen peroxide producing system, or hypohalites such as chlorine bleaches like hypochlorite, may also be used. Preferred oxygen bleaches include alkali metal salts of perborate, percarbonate, and mixtures thereof.

Oxygen bleaches deliver "available oxygen" (AvO) or "active oxygen" which is typically measurable by standard methods such as iodide/thiosulfate and/or ceric sulfate titration. See the well-known work by Swern, or Kirk Othmer's Encyclopedia of Chemical Technology under "Bleaching Agents". When the oxygen bleach is a peroxygen compound, it contains -O-O- linkages with one O in each such linkage being "active". AvO content of such an oxygen bleach compound, usually expressed as a percent, is equal to 100 * the number of active oxygen atoms * (16 / molecular weight of the oxygen bleach compound).

Preferred oxygen bleaches, as noted, include the peroxohydrates, sometimes known as peroxyhydrates or peroxohydrates. These are organic or, more commonly, inorganic salts capable of releasing hydrogen peroxide readily. They include types in which hydrogen peroxide is present as a true crystal hydrate, and types in which hydrogen peroxide is incorporated covalently and is released chemically, for example by hydrolysis. Typically, peroxohydrates deliver hydrogen peroxide readily enough that it can be extracted in measurable amounts into the ether phase of an ether/water mixture. Peroxohydrates are characterized in that they fail to give the Riesenfeld reaction, in contrast to certain other oxygen bleach types described hereinafter. Peroxohydrates are the most common examples of "hydrogen peroxide source" materials and include the perborates, percarbonates, perphosphates, and persilicates. Other materials which serve to produce or release hydrogen peroxide are, of course, useful. Mixtures of two or more peroxohydrates can be used, for example when it is desired to exploit differential solubility. Suitable peroxohydrates include sodium carbonate peroxyhydrate and equivalent commercial "percarbonate" bleaches, and any of the so-called sodium perborate hydrates, the "tetrahydrate" and "monohydrate" being preferred; though sodium pyrophosphate peroxyhydrate can be used. Many such peroxohydrates are available in processed forms with coatings, such as of silicate and/or borate and/or waxy materials and/or surfactants, or have particle geometries, such as compact spheres, which

improve storage stability. By way of organic peroxyhydrates, urea peroxyhydrate can also be useful herein.

Percarbonate bleach includes, for example, dry particles having an average particle size in the range from about 500 micrometers to about 1,000 5 micrometers, not more than about 10% by weight of said particles being smaller than about 200 micrometers and not more than about 10% by weight of said particles being larger than about 1,250 micrometers. Percarbonates and perborates are widely available in commerce, for example from FMC, Solvay and Tokai Denka.

Common oxygen bleaches of the peroxygen type include hydrogen peroxide, inorganic peroxyhydrates, organic peroxyhydrates and the organic peroxyacids, including hydrophilic and hydrophobic mono- or di- peroxyacids. These can be peroxycarboxylic acids, peroxyimidic acids, amidoperoxycarboxylic acids, or their salts including the calcium, magnesium, or mixed-cation salts. 10
15 Peracids of various kinds can be used both in free form and as precursors known as "bleach activators" or "bleach promoters" which, when combined with a source of hydrogen peroxide, perhydrolyze to release the corresponding peracid.

Also useful herein as oxygen bleaches are the inorganic peroxides such as Na₂O₂, superoxides such as KO₂, organic hydroperoxides such as cumene 20 hydroperoxide and t-butyl hydroperoxide, and the inorganic peroxyacids and their salts such as the peroxydisulfuric acid salts, especially the potassium salts of peroxydisulfuric acid and, more preferably, of peroxyomonosulfuric acid including the commercial triple-salt form sold as OXONE™ by DuPont and also any equivalent commercially available forms such as CUROX™ from Akzo or 25 CAROAT™ from Degussa. Certain organic peroxides, such as dibenzoyl peroxide, may be useful, especially as additives rather than as primary oxygen bleach.

Organic percarboxylic acids useful herein as the oxygen bleach include magnesium monoperoxyphthalate hexahydrate, available from Interox, *m*-chloro 30 perbenzoic acid and its salts, 4-nonylamino-4-oxoperoxybutyric acid and diperoxydodecanedioic acid and their salts. Such bleaching agents are disclosed in U.S. Patent 4,483,781 to Hartman, issued November 20, 1984, U.S. Patent Application 740,446 to Burns et al., filed June 3, 1985, European Patent Application 0,133,354 to Banks et al., published February 20, 1985, and U.S. 35 Patent 4,412,934 to Chung and Spadini, issued November 1, 1983. Examples of

such oxygen bleaches also include 6-nonylamino-6-oxoperoxycaproic acid (NAPAA) as described in U.S. Patent 4,634,551 to Hardy and Ingram, issued January 6, 1987, and include those having formula HO-O-C(O)-R-Y wherein R is an alkylene or substituted alkylene group containing from 1 to about 22 carbon atoms or a phenylene or substituted phenylene group, and Y is hydrogen, halogen, alkyl, aryl or -C(O)-OH or -C(O)-O-OH.

5 Bleaching enzymes, such as peroxidase, may also be used as an oxygen bleach.

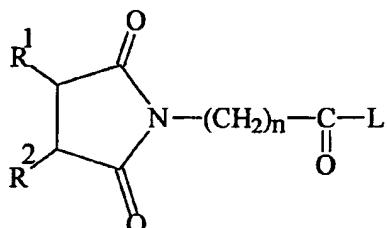
10 2. Bleach activators
The encapsulated detergent particle of the present invention may contain a bleach activator. With respect to the above bleach activator structure RC(O)L, the atom in the leaving group connecting to the peracid-forming acyl moiety R(C)O- is most typically O or N. Bleach activators can have non-charged, positively or negatively charged peracid-forming moieties and/or noncharged, 15 positively or negatively charged leaving groups. One or more peracid-forming moieties or leaving-groups can be present. See, for example, U.S. 5,595,967 to Kellett, et al., issued January 21, 1997, U.S. 5,561,235 to Burns, et al., issued October 1, 1996, U.S. 5,560,862 to Burns, et al., issued October 1, 1996 or the bis-(peroxy-carbonic) system of U.S. 5,534,179 to Kellett, et al., issued July 9, 20 1996.

25 Cationic bleach activators include quaternary carbamate-, quaternary carbonate-, quaternary ester- and quaternary amide- types, delivering a range of cationic peroxyimidic, peroxycarbonic or peroxycarboxylic acids to the wash. An analogous but non-cationic palette of bleach activators is available when quaternary derivatives are not desired. In more detail, cationic activators include 2-(N,N,N-trimethyl ammonium) ethyl-4-sulphophenyl carbonate-(SPCC); N-octyl,N,N-dimethyl-N 10-carbophenoxy decyl ammonium chloride-(ODC); 3-(N,N,N-trimethyl ammonium) propyl sodium-4-sulphophenyl carboxylate; and N,N,N-trimethyl ammonium toluyloxy benzene sulfonate. Other nitrile types such 30 as 3,5-dimethoxybenzonitrile and 3,5-dinitrobenzonitrile can also be used.

Preferred bleach activators include N,N,N'N'-tetraacetyl ethylene diamine (TAED) or any of its close relatives including the triacetyl or other unsymmetrical derivatives. TAED and the acetylated carbohydrates such as glucose pentaacetate and tetraacetyl xylose are preferred hydrophilic bleach activators.

Depending on the application, acetyl triethyl citrate, a liquid, also has some utility, as does phenyl benzoate.

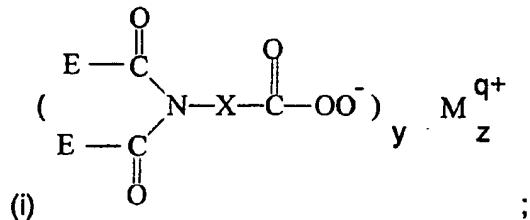
Preferred hydrophobic bleach activators include sodium nonanoyloxybenzene sulfonate (NOBS or SNOBS), substituted amide types described in detail hereinafter, such as activators related to NAPAA, and activators related to certain imidoperoacid bleaches, for example as described in U.S. Patent 5,061,807 to Gethoffer, et al., issued October 29, 1991 and assigned to Hoechst Aktiengesellschaft of Frankfurt, Germany. Japanese Laid-Open Patent Application (Kokai) No. 4-28799 to Yamada, et al., published January 31, 1992 for example describes a bleaching agent and a bleaching detergent composition comprising an organic peracid precursor described by a general formula and illustrated by compounds which may be summarized more particularly as conforming to the formula:



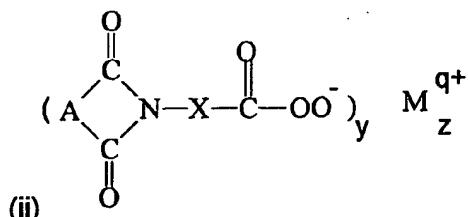
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wherein L is sodium *p*-phenolsulfonate, R¹ is CH₃ or C₁₂H₂₅ and R² is H. Analogs of these compounds having any of the leaving-groups identified herein and/or having R¹ being linear or branched C₆-C₁₆ are also useful.

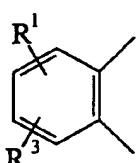
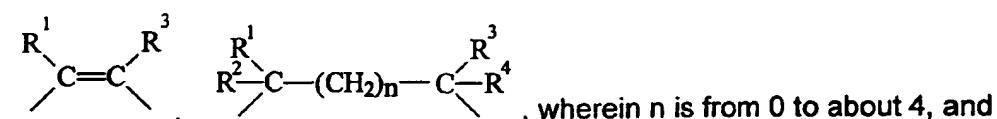
20 Another group of peracids and bleach activators herein are those derivable from acyclic imidoperoxycarboxylic acids and salts thereof of the formula:



25 cyclic imidoperoxycarboxylic acids and salts thereof of the formula:



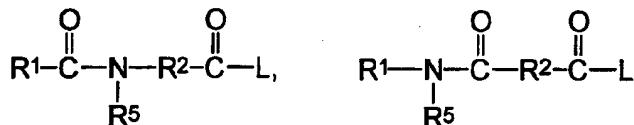
and (iii) mixtures of said compounds, (i) and (ii); wherein M is selected from hydrogen and bleach-compatible cations having charge q; and y and z are integers such that said compound is electrically neutral; E, A and X comprise hydrocarbyl groups; and said terminal hydrocarbyl groups are contained within E and A. The structure of the corresponding bleach activators is obtained by deleting the peroxy moiety and the metal and replacing it with a leaving-group L, which can be any of the leaving-group moieties defined elsewhere herein. In preferred embodiments, there are encompassed detergent compositions wherein, in any of said compounds, X is linear C₃-C₈ alkyl; A is selected from:



; wherein R¹ and E are said terminal hydrocarbyl groups, R², R³ and R⁴ are independently selected from H, C₁-C₃ saturated alkyl, and C₁-C₃ unsaturated alkyl; and wherein said terminal hydrocarbyl groups are alkyl groups comprising at least six carbon atoms, more typically linear or branched alkyl having from about 8 to about 16 carbon atoms.

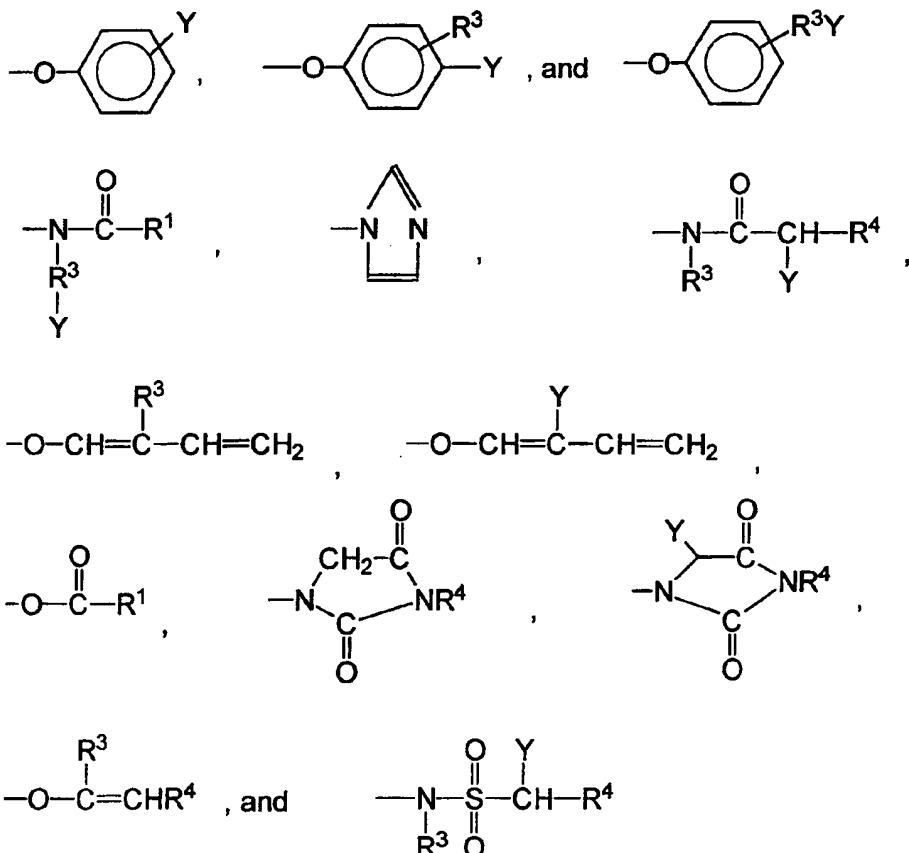
Other suitable bleach activators include sodium-4-benzoyloxy benzene sulfonate (SBOBS); sodium-1-methyl-2-benzoyloxy benzene-4-sulphonate; sodium-4-methyl-3-benzoyloxy benzoate (SPCC); trimethyl ammonium toluoyloxy-benzene sulfonate; or sodium 3,5,5-trimethyl hexanoyloxybenzene sulfonate (STHOBS).

Highly preferred bleach activators useful herein are amide-substituted and have either of the formulae:



or mixtures thereof, wherein R^1 is alkyl, aryl, or alkaryl containing from about 1 to about 14 carbon atoms including both hydrophilic types (short R^1) and hydrophobic types (R^1 is especially from about 8 to about 12), R^2 is alkylene, arylene or alkarylene containing from about 1 to about 14 carbon atoms, R^5 is H, or an alkyl, aryl, or alkaryl containing from about 1 to about 10 carbon atoms, and L is a leaving group.

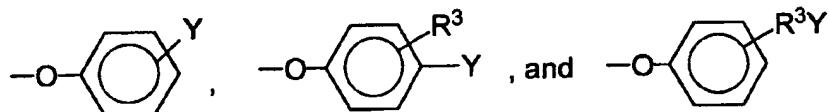
Preferred bleach activators include those of the formulae, for example the amide-substituted formulae, hereinabove, wherein R^1 , R^2 and R^5 are as defined for the corresponding peroxyacid and L is selected from the group consisting of:



and mixtures thereof, wherein R^1 is a linear or branched alkyl, aryl, or alkaryl group containing from about 1 to about 14 carbon atoms, R^3 is an alkyl chain

containing from 1 to about 8 carbon atoms, R⁴ is H or R³, and Y is H or a solubilizing group. These and other known leaving groups are, more generally, general suitable alternatives for introduction into any bleach activator herein. Preferred solubilizing groups include -SO₃⁻M⁺, -CO₂⁻M⁺, -SO₄⁻M⁺, -N^{+(R)4X-} and O-N(R³)₂, more preferably -SO₃⁻M⁺ and -CO₂⁻M⁺ wherein R³ is an alkyl chain containing from about 1 to about 4 carbon atoms, M is a bleach-stable cation and X is a bleach-stable anion, each of which is selected consistent with maintaining solubility of the activator. Melting-point reduction can be favored by incorporating branched, rather than linear alkyl moieties into the oxygen bleach or precursor.

Preferred bleach activators also include those of the above general formula wherein L is selected from the group consisting of:



wherein R³ is as defined above and Y is -SO₃⁻M⁺ or -CO₂⁻M⁺ wherein M is as defined above.

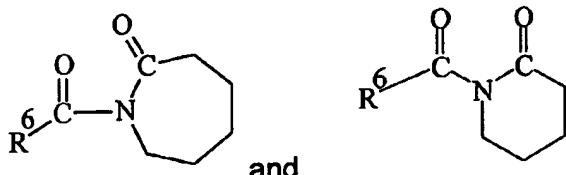
Preferred examples of bleach activators of the above formulae include:

(6-octanamidocaproyl)oxybenzenesulfonate,

(6-nonanamidocaproyl)oxybenzenesulfonate,

(6-decanamidocaproyl)oxybenzenesulfonate, and mixtures thereof.

Acyl lactam activators are very useful herein, especially the acyl caprolactams (see for example WO 94-28102 A to Burns, et al., published December 8, 1994) and acyl valerolactams (see) of the formulae:



wherein R⁶ is H, alkyl, aryl, alkoxyaryl, an alkaryl group containing from 1 to about 12 carbon atoms, or substituted phenyl containing from about 6 to about 18 carbons. Also useful are acyl caprolactams, including benzoyl caprolactam adsorbed into sodium perborate. In certain preferred embodiments of the invention, NOBS, lactam activators, imide activators or amide-functional activators, especially the more hydrophobic derivatives, are desirably combined with hydrophilic activators such as TAED, typically at weight ratios of

hydrophobic activator : TAED in the range of 1:5 to 5:1, preferably about 1:1. Other suitable lactam activators are alpha-modified, see WO 96-22350 A1 to Burekett, et al., published July 25, 1996. Lactam activators, especially the more hydrophobic types, are desirably used in combination with TAED, typically at weight ratios of amido-derived or caprolactam activators : TAED in the range of 1:5 to 5:1, preferably about 1:1. See also the bleach activators having cyclic amidine leaving-group disclosed in U.S. 5,552,556 to Burns, et al., issued September 3, 1996.

Nonlimiting examples of additional activators useful herein are to be found in U.S. 4,915,854 to Baker, et al., issued April 10, 1990, U.S. 4,412,934 to Chung and Spadini, issued November 1, 1983 and 4,634,551 to Hardy and Ingram, issued January 6, 1987. The hydrophobic activator nonanoyloxybenzene sulfonate (NOBS) and the hydrophilic tetraacetyl ethylene diamine (TAED) activator are typical, and mixtures thereof can also be used.

Additional activators useful herein include those of U.S. 5,545,349 to Itoh, et al., issued August 13, 1996. Examples include esters of an organic acid and ethylene glycol, diethylene glycol or glycerin, or the acid imide of an organic acid and ethylenediamine; wherein the organic acid is selected from methoxyacetic acid, 2-methoxypropionic acid, p-methoxybenzoic acid, ethoxyacetic acid, 2-ethoxypropionic acid, p-ethoxybenzoic acid, propoxyacetic acid, 2-propoxypropionic acid, p-propoxybenzoic acid, butoxyacetic acid, 2-butoxypropionic acid, p-butoxybenzoic acid, 2-methoxyethoxyacetic acid, 2-methoxy-1-methylethoxyacetic acid, 2-methoxy-2-methylethoxyacetic acid, 2-ethoxyethoxyacetic acid, 2-(2-ethoxyethoxy)propionic acid, p-(2-ethoxyethoxy)benzoic acid, 2-ethoxy-1-methylethoxyacetic acid, 2-ethoxy-2-methylethoxyacetic acid, 2-propoxyethoxyacetic acid, 2-propoxy-1-methylethoxyacetic acid, 2-propoxy-2-methylethoxyacetic acid, 2-butoxyethoxyacetic acid, 2-butoxy-1-methylethoxyacetic acid, 2-butoxy-2-methylethoxyacetic acid, 2-(2-methoxyethoxy)ethoxyacetic acid, 2-(2-methoxy-1-methylethoxy)ethoxyacetic acid, 2-(2-methoxy-2-methylethoxy)ethoxyacetic acid and 2-(2-ethoxyethoxy)ethoxyacetic acid.

3. Polymeric soil release agents

The encapsulated detergent particle of the present invention may contain a polymeric soil release agent, hereinafter "SRA".

Suitable SRAs include a sulfonated product of a substantially linear ester oligomer comprised of an oligomeric ester backbone of terephthaloyl and oxyalkyleneoxy repeat units and allyl-derived sulfonated terminal moieties covalently attached to the backbone, for example as described in U.S. 5 4,968,451, November 6, 1990 to Scheibel and Gosselink. Other SRA's include the nonionic end-capped 1,2-propylene/polyoxyethylene terephthalate polyesters of U.S. 4,711,730, December 8, 1987 to Gosselink et al.

SRA's also include: simple copolymeric blocks of ethylene terephthalate or propylene terephthalate with polyethylene oxide or polypropylene oxide terephthalate, such as METHOCEL from Dow, and METOLOSE SM100 and METOLOSE SM200, which are the trade names of methyl cellulose ethers manufactured by Shin-etsu Kagaku Kogyo KK.

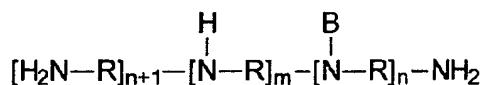
Suitable SRA's characterized by poly(vinyl ester) hydrophobe segments include graft copolymers of poly(vinyl ester), e.g., C₁-C₆ vinyl esters, preferably poly(vinyl acetate), grafted onto polyalkylene oxide backbones. Other SRA's are polyesters with repeat units containing 10-15% by weight of ethylene terephthalate together with 80-90% by weight of polyoxyethylene terephthalate derived from a polyoxyethylene glycol of average molecular weight 300-5,000. Commercial examples include ZELCON 5126 from DuPont and MILEASE T from ICI.

Another preferred SRA is an oligomer having empirical formula (CAP)₂(EG/PG)₅(T)₅(SIP)₁ which comprises terephthaloyl (T), sulfoisophthaloyl (SIP), oxyethyleneoxy and oxy-1,2-propylene (EG/PG) units and which is preferably terminated with end-caps (CAP), preferably modified isethionates, as in an oligomer comprising one sulfoisophthaloyl unit, 5 terephthaloyl units, oxyethyleneoxy and oxy-1,2-propyleneoxy units in a defined ratio, preferably about 0.5:1 to about 10:1, and two end-cap units derived from sodium 2-(2-hydroxyethoxy)-ethanesulfonate. Said SRA preferably further comprises from 0.5% to 20%, by weight of the oligomer, of a crystallinity-reducing stabilizer, for example an anionic surfactant such as linear sodium dodecylbenzenesulfonate or a member selected from xylene-, cumene-, and toluene- sulfonates or mixtures thereof, these stabilizers or modifiers being introduced into the synthesis vessel, all as taught in U.S. 5,415,807, to Gosselink, et al., issued May 16, 1995. Suitable monomers for the above SRA include Na-2-(2-

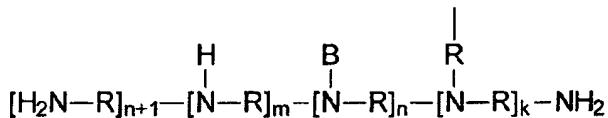
hydroxyethoxy)-ethanesulfonate, DMT, Na-dimethyl-5-sulfoisophthalate, EG and PG.

The detergent composition can optionally contain a polyamine soil release agent related to modified polyamines. See U.S. 5,565,145 issued October 15, 5 1996 to Watson et al.

The linear or non-cyclic polyamine backbones that comprise the modified polyethyleneimine polymers of the present invention have the general formula:



10 said backbones prior to subsequent modification, comprise primary, secondary and tertiary amine nitrogens connected by R "linking" units. The cyclic polyamine backbones comprising the modified polyethyleneimine polymers of the present invention have the general formula:



15 said backbones prior to subsequent modification, comprise primary, secondary and tertiary amine nitrogens connected by R "linking" units.

The preferred polyamines include generally polyalkyleneamines (PAA's), polyalkyleneimines (PAI's), preferably polyethyleneamine (PEA's), 20 polyethyleneimines (PEI's), or PEA's or PEI's connected by moieties having longer R units than the parent PAA's, PAI's, PEA's or PEI's. A common polyalkyleneamine (PAA) is tetrabutylpentamine. PEA's are obtained by reactions involving ammonia and ethylene dichloride, followed by fractional distillation. The common PEA's obtained are triethylenetetramine (TETA) and 25 teraethylenepentamine (TEPA). Above the pentamines, i.e., the hexamines, heptamines, octamines and possibly nonamines, the cogenerically derived mixture does not appear to separate by distillation and can include other materials such as cyclic amines and particularly piperazines. There can also be present cyclic amines with side chains in which nitrogen atoms appear. See U.S. 30 Patent 2,792,372, Dickinson, issued May 14, 1957, which describes the preparation of PEA's.

Particularly suitable polymeric polycarboxylates can be derived from acrylic acid. Such acrylic acid-based polymers which are useful herein are the

water-soluble salts of polymerized acrylic acid. The average molecular weight of such polymers in the acid form preferably ranges from about 2,000 to 10,000, more preferably from about 4,000 to 7,000 and most preferably from about 4,000 to 5,000. Water-soluble salts of such acrylic acid polymers can include, for 5 example, the alkali metal, ammonium and substituted ammonium salts. Soluble polymers of this type are known materials. Use of polyacrylates of this type in detergent compositions has been disclosed, for example, in Diehl, U.S. Patent 3,308,067, issued march 7, 1967.

Acrylic/maleic-based copolymers may also be used as an SRA. Such 10 materials include the water-soluble salts of copolymers of acrylic acid and maleic acid. The average molecular weight of such copolymers in the acid form preferably ranges from about 2,000 to 100,000, more preferably from about 5,000 to 75,000, most preferably from about 7,000 to 65,000. The ratio of acrylate to maleate segments in such copolymers will generally range from about 15 30:1 to about 1:1, more preferably from about 10:1 to 2:1. Water-soluble salts of such acrylic acid/maleic acid copolymers can include, for example, the alkali metal, ammonium and substituted ammonium salts. Soluble acrylate/maleate copolymers of this type are known materials which are described in European Patent Application No. published December 15, 1982, as well as in , published 20 September 3, 1986, which also describes such polymers comprising hydroxypropylacrylate. Still other useful dispersing agents include the maleic/acrylic/vinyl alcohol terpolymers.

4. Enzymes

The encapsulated detergent particle of the present invention may contain 25 an enzyme or mixtures of enzymes. Preferred deterutive enzymes are hydrolases such as proteases, amylases and lipases. Preferred enzymes for laundry purposes include, but are not limited to, proteases, cellulases, lipases and peroxidases. Highly preferred for automatic dishwashing are amylases and/or proteases, including both current commercially available types and improved 30 types which, though more and more bleach compatible though successive improvements, have a remaining degree of bleach deactivation susceptibility.

Suitable examples of proteases are the subtilisins which are obtained from particular strains of *B. subtilis* and *B. licheniformis*. One suitable protease is obtained from a strain of *Bacillus*, having maximum activity throughout the pH 35 range of 8-12, developed and sold as ESPERASE® by Novo Industries A/S of

Denmark, hereinafter "Novo". The preparation of this enzyme and analogous enzymes is described in to Novo. Other suitable proteases include ALCALASE® and SAVINASE® from Novo and MAXATASE® from International Bio-Synthetics, Inc., The Netherlands; as well as Protease A and Protease B as 5 disclosed in EP 130,756 A to Bott, published January 9, 1985 and Protease B as disclosed in EP 303,761 B, to Post, et al., issued September 9, 1992.

In more detail, an especially preferred protease, referred to as "Protease D" is a carbonyl hydrolase variant having an amino acid sequence not found in nature, which is derived from a precursor carbonyl hydrolase by substituting a 10 different amino acid for a plurality of amino acid residues at a position in said carbonyl hydrolase equivalent to position +76, preferably also in combination with one or more amino acid residue positions equivalent to those selected from the group consisting of +99, +101, +103, +104, +107, +123, +27, +105, +109, +126, +128, +135, +156, +166, +195, +197, +204, +206, +210, +216, +217, 15 +218, +222, +260, +265, and/or +274 according to the numbering of *Bacillus amyloliquefaciens* subtilisin, as described in U.S. Patent 5,679,630 to A. Baeck, et al, issued October 21, 1997, entitled "Protease-Containing Cleaning Compositions," and U.S. Patent 5,677,272 to C. Ghosh, et al, issued October 14, 1997, entitled "Bleaching Compositions Comprising Protease Enzymes."

20 Amylases suitable herein, especially for, but not limited to automatic dishwashing purposes, include, for example, -amylases described in GB 1,296,839 to Outtrup H, et al., published November 22, 1972 to Novo; RAPIDASE®, International Bio-Synthetics, Inc. and TERMAMYL®, Novo. FUNGAMYL® from Novo is especially useful. Engineering of enzymes for 25 improved stability, e.g., oxidative stability, is known. See, for example J. Biological Chem., Vol. 260, No. 11, June 1985, pp. 6518-6521. Certain preferred embodiments of the present compositions can make use of amylases having improved stability in detergents such as automatic dishwashing types, especially improved oxidative stability as measured against a reference-point of 30 TERMAMYL® in commercial use in 1993. Stability can be measured using any of the art-disclosed technical tests. See, for example, references disclosed in WO 9402597 to Bisgard-Frantzen and Svendsen, published February 3, 1994. Stability-enhanced amylases can be obtained from Novo or from Genencor International. One class of highly preferred amylases herein have the 35 commonality of being derived using site-directed mutagenesis from one or more

of the *Bacillus* amylases, especially the *Bacillus* -amylases, regardless of whether one, two or multiple amylase strains are the immediate precursors. Oxidative stability-enhanced amylases vs. the above-identified reference amylase are preferred for use, especially in bleaching, more preferably oxygen bleaching, as distinct from chlorine bleaching, detergent compositions herein.

Cellulases usable herein include both bacterial and fungal types, preferably having a pH optimum between 5 and 9.5. U.S. 4,435,307, Barbesgaard et al, March 6, 1984, discloses suitable fungal cellulases from *Humicola insolens* or *Humicola* strain DSM1800 or a cellulase 212-producing fungus belonging to the genus *Aeromonas*, and cellulase extracted from the hepatopancreas of a marine mollusk, *Dolabella Auricula Solander*. CAREZYME® and CELLUZYME® (Novo) are especially useful. See also WO 9117243 to Hagen, et al., published November 14, 1991 as to Novo.

Suitable lipase enzymes for detergent usage include those produced by microorganisms of the *Pseudomonas* group, such as *Pseudomonas stutzeri* ATCC 19.154, as disclosed in GB 1,372,034 to Dijk and Berg, published October 30, 1974. See also lipases in Japanese Patent Application 53,20487 to Inugai, published February 24, 1978. This lipase is available from Amano Pharmaceutical Co. Ltd., Nagoya, Japan, under the trade name Lipase P "Amano," or "Amano-P." Other suitable commercial lipases include Amano-CES, lipases ex *Chromobacter viscosum*, e.g. *Chromobacter viscosum* var. *lipolyticum* NRRLB 3673 from Toyo Jozo Co., Tagata, Japan; *Chromobacter viscosum* lipases from U.S. Biochemical Corp., U.S.A. and Disoynth Co., The Netherlands, and lipases ex *Pseudomonas gladioli*. LIPOLASE® enzyme derived from *Humicola lanuginosa* and commercially available from Novo, see also EP 341,947 to Cornelissen, et al., issued August 31, 1994, is a preferred lipase for use herein.

In spite of the large number of publications on lipase enzymes, only the lipase derived from *Humicola lanuginosa* and produced in *Aspergillus oryzae* as host has so far found widespread application as additive for fabric washing products. It is available from Novo Nordisk under the tradename Lipolase™, as noted above.

Cutinase enzymes suitable for use herein are described in WO 8809367A to Kolattukudy, et al., published December 1, 1988 to Genencor.

Peroxidase enzymes may be used in combination with oxygen sources, e.g., percarbonate, perborate, hydrogen peroxide, etc., for "solution bleaching" or prevention of transfer of dyes or pigments removed from substrates during the wash to other substrates present in the wash solution. Known peroxidases 5 include horseradish peroxidase, ligninase, and haloperoxidases such as chloro- or bromo-peroxidase. Peroxidase-containing detergent compositions are disclosed in, October 19, 1989 to Novo and WO 8909813 A to Damhus, et al., published October 19, 1989 to Novo.

5. Fabric Softening Agents

10 The encapsulated detergent particle of the present invention may contain a fabric softening agent. Various through-the-wash fabric softeners, especially the impalpable smectite clays of U.S. Patent 4,062,647, Storm and Nirschl, issued December 13, 1977, as well as other softener clays known in the art to provide fabric softener benefits concurrently with fabric cleaning. Among suitable 15 cationic softeners are the conventional substantially water-insoluble quaternary ammonium compounds, and C₈₋₂₅ alkyl imidazolinium salts. Representative examples of quaternary softeners include ditallow dimethyl ammonium chloride; ditallow dimethyl ammonium methyl sulfate; dihexadecyl dimethyl ammonium chloride; di(hydrogenated tallow) dimethyl ammonium chloride; dioctadecyl 20 dimethyl ammonium chloride; dieicosyl dimethyl ammonium chloride; didocosyl ammonium chloride; di(hydrogenated tallow) dimethyl ammonium methyl sulfate; dihexadecyl dimethyl ammonium chloride; di(coconutalkyl) dimethyl ammonium chloride. Other representative examples include lauryl trimethyl ammonium bromide, lauryl dimethyl benzyl ammonium chloride, myristyl dimethyl ethyl 25 ammonium bromide, cetyl trimethyl ammonium bromide, behenyl trimethyl ammonium methosulfate oleyl methyl diethyl ammonium chloride, cetyl stearyl or oleyl pyridinium chloride, behenyl pyridinium bromide, stearyl methyl morpholinium chloride, stearyl or oleyl ethyl or propyl morpholinium chloride. Clay softeners can be used in combination with amine and cationic softeners as 30 disclosed, for example, in U.S. Patent 4,375,416 to Crisp et al., March 1, 1983 and U.S. Patent 4,291,071 to Harris et al., issued September 22, 1981.

6. Suds Suppressors

35 The encapsulated detergent particle of the present invention may contain a suds suppressor. One category of suds suppressor of particular interest encompasses monocarboxylic fatty acid and soluble salts therein. See U.S. Patent 2,954,347,

issued September 27, 1960 to Wayne. The monocarboxylic fatty acids and salts thereof used as suds suppressor typically have hydrocarbyl chains of 10 to about 24 carbon atoms, preferably 12 to 18 carbon atoms. Suitable salts include the alkali metal salts such as sodium, potassium, and lithium salts, and ammonium 5 and alkanolammonium salts.

The detergent compositions herein may also contain non-surfactant suds suppressors. These include, for example: high molecular weight hydrocarbons such as paraffin, fatty acid esters (e.g., fatty acid triglycerides), fatty acid esters of monovalent alcohols, aliphatic C₁₈-C₄₀ ketones (e.g., stearone), etc. Other 10 suds inhibitors include N-alkylated amino triazines such as tri- to hexa-alkylmelamines or di- to tetra-alkyldiamine chlorotriazines formed as products of cyanuric chloride with two or three moles of a primary or secondary amine containing 1 to 24 carbon atoms, propylene oxide, and monostearyl phosphates containing 15 such as monostearyl alcohol phosphate ester and monostearyl di-alkali metal (e.g., K, Na, and Li) phosphates and phosphate esters.

Another preferred category of non-surfactant suds suppressors comprises silicone suds suppressors. This category includes the use of polyorganosiloxane oils, such as polydimethylsiloxane, dispersions or emulsions of polyorganosiloxane oils or resins, and combinations of polyorganosiloxane with 20 silica particles wherein the polyorganosiloxane is chemisorbed or fused onto the silica.

An exemplary silicone based suds suppressor for use herein is a suds suppressing amount of a suds controlling agent consisting essentially of:

- (i) polydimethylsiloxane fluid having a viscosity of from about 20 cs. to 25 about 1,500 cs. at 25°C;
- (ii) from about 5 to about 50 parts per 100 parts by weight of (i) of siloxane resin composed of (CH₃)₃SiO_{1/2} units of SiO₂ units in a ratio of from (CH₃)₃ SiO_{1/2} units and to SiO₂ units of from about 0.6:1 to about 1.2:1; and
- 30 (iii) from about 1 to about 20 parts per 100 parts by weight of (i) of a solid silica gel.

In the preferred silicone suds suppressor used herein, the solvent for a continuous phase is made up of certain polyethylene glycols or polyethylene-polypropylene glycol copolymers or mixtures thereof (preferred), or

polypropylene glycol. The primary silicone suds suppressor is branched/crosslinked and preferably not linear.

The silicone suds suppressor herein preferably comprises polyethylene glycol and a copolymer of polyethylene glycol/polypropylene glycol, all having an average molecular weight of less than about 1,000, preferably between about 100 and 800. The polyethylene glycol and polyethylene/polypropylene copolymers herein have a solubility in water at room temperature of more than about 2 weight %, preferably more than about 5 weight %.

The preferred solvent herein is polyethylene glycol having an average molecular weight of less than about 1,000, more preferably between about 100 and 800, most preferably between 200 and 400, and a copolymer of polyethylene glycol/polypropylene glycol, preferably PPG 200/PEG 300. Preferred is a weight ratio of between about 1:1 and 1:10, most preferably between 1:3 and 1:6, of polyethylene glycol:copolymer of polyethylene-polypropylene glycol.

The preferred silicone suds suppressors used herein do not contain polypropylene glycol, particularly of 4,000 molecular weight. They also preferably do not contain block copolymers of ethylene oxide and propylene oxide, like PLURONIC L101.

Other suds suppressors useful herein comprise the secondary alcohols (e.g., 2-alkyl alkanols) and mixtures of such alcohols with silicone oils, such as the silicones disclosed in U.S. The secondary alcohols include the C₆-C₁₆ alkyl alcohols having a C₁-C₁₆ chain. A preferred alcohol is 2-butyl octanol, which is available from Condea under the trademark ISOFOL 12. Mixtures of secondary alcohols are available under the trademark ISALCHEM 123 from Enichem. Mixed suds suppressors typically comprise mixtures of alcohol + silicone at a weight ratio of 1:5 to 5:1.

7. Bactericides and bacteriostats

The encapsulated detergent particle of the present invention may contain bactericides and/or bacteriostats. The bactericides and bacteriostats are preferably highly soluble in alkaline conditions. Representative examples include Triclosan and Triclocarban. Cationic surfactants, like ditallow dimethyl diammonium chloride, may also be used as a bacteriostat and/or bactericide. Other examples include natural alcohols. A preferred alcohol is Geraniol.

35 B. Coating material

The encapsulated detergent particle of the present invention contains a coating material. Ideally the coating material is selected so that it remains substantially water insoluble during the beginning of the main washing cycle, while becoming more soluble as the wash progresses, especially during rinsing.

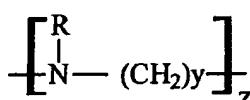
- 5 A preferred coating material is one that has a melting point no less than 30°C, and a pKa (or protonation constant) of no more than 12 and no less than 7.0, preferably between 11.5 and 8.0, most preferably between 10.5 and 9.0. The protonation constant K is defined as $K = -\log [H^+Z]/[H^+]^*[Z]$ where Z is the selected chemical and $[H^+]$ is the hydrogen ion concentration in solution. The
- 10 preferred material is selected so that during the early part of the wash where the wash solution pH is controlled by the detergent at around 10, the coating material is substantially neutral without a charge, and remains relatively water insoluble. As the wash progresses, the wash pH will decrease either by soil dissolution or rinse water addition, the coating material will then be protonated to
- 15 become positively charged, and more water soluble. This will then allow the breaking down of the coating shell to allow the release of the beneficial agents contained within.

A preferred class of coating materials include amines, waxes, Schiff base compounds, and mixtures thereof.

- 20 Chemicals of amines are usually represented by the general formula of R-N(R')-R'', and its oligomeric forms, i.e., Rx-N(Ry)-Rz-[N(Rw)-Ru]n-N(Rv)-Rs, where Rx, Ry, Rz, Rw, Ru, Rv and Rs can be differently substituted alkyl functional groups, selected from, but not limited to, linear methyl through octadecyl, branched hydrocarbyls, hydrocarbyl chains containing other functional
- 25 groups such as hydroxyls, ethers, double bonds and triple bonds, etc.

- Preferred amines include amines other than primary fatty amines. Such amines include polymeric aminofunctional homopolymers and/or copolymers and/or terpolymers. The preferred amines typically do not have exclusively primary amino functionality; rather, they typically contain secondary and/or tertiary amino groups, optionally with primary amino moieties also included.

A preferred coating material is a polymeric amine having the formula



wherein y is an integer of from 1 to 4, z is an integer greater than 1, R is selected from the group consisting of hydrogen and alkyl, hydroxy alkyl, and alkanoyl

groups containing from about 1 to about 22 carbon atoms, from about 5% to about 100%, more preferably from about 10% to about 50%, of the nitrogen atoms in the polymeric amine being substituted with alkyl or alkanoyl groups containing 1 to 22 carbon atoms. The coatings can have any degree of polymerization, but are preferably in the range of from 2 to 50,000, more preferably in the range of from 20 to 10,000 monomer units per molecule.

5 A specific example would be N1-hydroxyethyl, N2,N3,dimethyl,N3-dodecyl diethylenetriamine.

10 The presence of the alkyl substitutions on the nitrogen is deemed important as it improves the filming property of the coating material. Alternatively, waxes can be used as a co-melted blend to improve the filming property. The preferred preferred ratio of wax to amines is no more than 50% wax.

15 An alternate class of chemicals are Schiff bases, i.e., the adduct of amines with aldehydes. These compounds are chemically stable at higher pH while able to break down to its components at lower pHs. However, most common Schiff bases decompose at pH lower than the preferred pH of the current invention. Only selected types that can break down at the pH of the current invention would be suitable. While not meant to be limited by theory, 20 once such example of a preferred Schiff base is formed between p-amino benzoate with an alkyl aldehyde. The alkyl chainlength on the aldehyde can be adjusted so to provide the desirable melting point characteristics.

A preferred example is the Schiff base made by fusing p-amino benzoate with a n-dodecyl aldehyde.

25 The coating material layer will typically range from about 1 to about 500, preferably from about 5 to about 100 microns thick. In a preferred embodiment, the coating layer is of a substantially uniform thickness throughout. In another preferred embodiment, the particle is substantially completely coated with at least one coating material layer. Preferably, the encapsulated detergent particle 30 has from about 1 to about 5 coating material layers, preferably from about 1 to about 2 coating layers.

The coating material is typically present at a weight ratio of coating material to the detergent active of from 1:99 to 75:25, preferably from 2:98 to 50:50. By weight, preferably, the coating material is from about 0.5% to 300% of

the detergent active, more preferably from about 5% to about 100%, and even more preferably from about 10% to about 50%.

Other detergents could be added into the coating material. In addition, the specific coating layer of the present invention may be used in conjunction with other known coating layers. For example, commercially available percarbonate may sometimes be coated. The coating material of the present invention may also be used as an additional layer of coating.

The coating material may be applied by conventional methods. The encapsulated particle of the invention may be made by conventional encapsulation processes wherein an active ingredient is coated by a coating layer. A preferred process useful herein for coating the particle is a fluidized bed process, such as the Wurster coating process. In a fluidized bed process, the active ingredient is fluidized, and the coating layer is sprayed onto the active ingredient, as it passes by the sprayer(s). The sprayer(s) may be located on the top, bottom and/or the sides of the fluidizing equipment. Co-current, counter-current, from the side, or a combination of these spraying configurations are available. A bottom spray configuration is preferred, because it typically provides more effective spraying.

The Wurster coating process utilizes an enclosed container which contains both a fluidizing mechanism and a spraying mechanism. When the particles are added to the container, they circulate in a given direction. The coating layer is applied by spraying it onto the particles as they pass by the spraying mechanism. In the Wurster process, the particle passes through the spray in a co-current direction.

Another conventional method for applying coating material involves agglomeration. Any conventional agglomeration/mixer may be used including but not limited to pan, rotary drum and vertical blender types. Molten coating compositions may also be applied either by being poured onto, or spray atomized onto a moving bed of detergent active source.

The improved coating material of the present invention may be sprayed onto the active ingredient as a molten liquid coating layer, in order to form an encapsulated particle. In such a process, special care needs to be taken to reduce or prevent high temperatures which could destabilize the active ingredient. Many oxygen bleaches, for example, may be destabilized by high temperatures. When coating such an oxygen bleach, the temperature of the

molten liquid coating layer should be less than about 100 °C, preferably less than 90 °C to avoid destabilizing the oxygen bleach. However, the melting temperature of the improved coating material is lower than comparable coating materials which lack an amine oxide. Accordingly, the molten liquid coating material of the current invention may be sprayed onto the active ingredient at a lower temperature, and is therefore less likely to destabilize the active ingredient.

5 **C. Detergent Compositions**

The encapsulated detergent particles of the present invention may be used in formulating detergent bar compositions. The encapsulated detergent particles are preferably used in amounts from about 0.01% to about 20%, more preferably from about 0.1% to about 10%, by weight of the bar composition.

10 The following describes various detergents components which are preferred in the detergent compositions of the present invention. As stated above, all of the detergent actives listed above which could be used in the encapsulated detergent particle may also be used as ingredients in the detergent composition, independent of the fact that the same detergent active or combinations of the detergent actives are used in the encapsulated detergent particle.

15 **Detergent Components**

20 **1. Detergent surfactants**

The detergent composition may also comprises a detergent surfactant, or otherwise also known as detergent actives or detergent surfactants. Typical detergent actives include anionic, nonionic, cationic, zwitterionic, and amphoteric surfactants. Detergent actives can be included at levels of from about 5% to about 60%, preferably from about 15% to about 30%, and more preferably from 25 about 20% to about 25%, by weight of the total bar composition. The detergent active can be either an acid form, neutralized form, or mixtures thereof.

Anionic synthetic detergent surfactants which are suitable for use herein include the water-soluble salts, preferably the alkali metal, ammonium and alkyldiammonium salts of organic sulfuric reaction products having in their molecular structure an alkyl group containing from about 10 to about 20 carbon atoms and a sulfonic acid or sulfuric acid ester group. (Included in the term "alkyl" is the alkyl portion of acyl groups.) Examples of this group of synthetic surfactants are the sodium and potassium alkyl sulfates, especially those obtained by sulfating the higher alcohols (C₈-18 carbon atoms) such as those produced by reducing the glycerides of tallow or coconut oil; and the sodium and

potassium alkylbenzene sulfonates in which the alkyl group contains from about 9 to about 15 carbon atoms, in straight chain or branched chain configuration, e.g., those of the type described in U.S. Patents 2,220,099 and 2,477,383. Especially valuable are linear straight chain alkylbenzene sulfonates in which the 5 average number of carbon atoms in the alkyl group is from about 10 to 18, abbreviated as C10-18 LAS. The alkali metal salts, particularly the sodium salts of these surfactants are preferred. Alkylbenzene sulfonates and processes for making them are disclosed in U.S. Patent Nos. 2,220,099 and 2,477,383.

Mixtures of the above types of anionic surfactants are preferred. 10 Specifically, preferred anionic surfactants are C10-18 linear alkyl benzene sulfonates, C10-18 alkyl sulfates, and mixtures thereof. One preferred composition comprises from about 10% to about 30% LAS, by weight of the total bar composition for a primarily LAS-surfactant based bar. Another preferred composition comprises a mixture of LAS:alkyl sulfate in a ratio of from about 15 10:90 to about 50:50, preferably from about 20:80 to about 40:60.

Other anionic synthetic surfactants suitable for use herein are the sodium alkyl glyceryl ether sulfonates, especially those ethers of higher alcohols derived from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfonates and sulfates. Preparation of alkyl glyceryl ether sulfonates are 20 described in detail in U.S. Pat. 3,024,273, Whyte et al., issued March 6, 1962.

In addition, suitable anionic synthetic surfactants include the water-soluble salts of esters of alpha-sulfonated fatty acids containing from about 6 to 20 carbon atoms in the fatty acid group and from about 1 to 10 carbon atoms in the ester group; water-soluble salts of 2-acyloxyalkane-1-sulfonic acids containing 25 from about 2 to 9 carbon atoms in the acyl group and from about 9 to about 23 carbon atoms in the alkane moiety; water-soluble salts of olefin and paraffin sulfonates containing from about 12 to 20 carbon atoms; and beta-alkyloxy alkane sulfonates containing from about 1 to 3 carbon atoms in the alkyl group and from about 8 to 20 carbon atoms in the alkane moiety.

30 Amine Oxides are excellent co-surfactants that may be used in alone or in addition to other detergent actives. Preferred types are C12-C18 amine oxides, preferably C14. If included, the level of amine oxide in the final bar composition is from about 1% to about 10%, preferably, from about 2% to about 5%.

Other anionic synthetic surfactants suitable for use herein are sodium 35 alkyl glyceryl ether sulfonates, especially those ethers of higher alcohols derived

from tallow and coconut oil; sodium coconut oil fatty acid monoglyceride sulfonates and sulfates. Preparation of alkyl glyceryl ether sulfonates are described in detail in U.S. Pat. 3,024,273, Whyte et al., issued March 6, 1962.

In addition, optional anionic synthetic surfactants include the water-soluble
5 salts of esters of alpha-sulfonated fatty acids containing from about 6 to 20 carbon atoms in the fatty acid group and from about 1 to 10 carbon atoms in the ester group; water-soluble salts of 2-acyloxyalkane-1-sulfonic acids containing from about 2 to 9 carbon atoms in the acyl group and from about 9 to about 23 carbon atoms in the alkane moiety; water-soluble salts of olefin and paraffin
10 sulfonates containing from about 12 to 20 carbon atoms; and beta-alkyloxy alkane sulfonates containing from about 1 to 3 carbon atoms in the alkyl group and from about 8 to 20 carbon atoms in the alkane moiety.

In addition, a hydrotrope, or mixture of hydrotropes, may be present in the laundry detergent bar. Preferred hydrotropes include the alkali metal, preferably
15 sodium, salts of toluene sulfonate, xylene sulfonate, cumene sulfonate, sulfosuccinate, and mixtures thereof. Preferably, the hydrotrope is added to the linear alkyl benzene sulfonic acid prior to its neutralization. The hydrotrope, if present, will preferably be present at from about 0.5% to about 5% of the laundry detergent bar.

20 **2. Builders**

Detergent builders may be included in the compositions herein to assist in controlling mineral hardness. Inorganic as well as organic builders can be used. Builders are typically used in fabric laundering compositions to assist in the removal of particulate soils.

25 The level of builder can vary widely depending upon the end use of the composition and its desired physical form. When present, the compositions will typically comprise at least about 1% builder. Bar formulations typically comprise from about 10% to about 80%, more typically from about 15% to about 50% by weight, of the detergent builder. Lower or higher levels of builder, however, are
30 not meant to be excluded.

Inorganic or P-containing detergent builders include, but are not limited to, the alkali metal, ammonium and alkanolammonium salts of polyphosphates (exemplified by the tripolyphosphates, pyrophosphates, and glassy polymeric meta-phosphates), phosphonates, phytic acid, silicates, carbonates (including
35 bicarbonates and sesquicarbonates), sulphates, and aluminosilicates. However,

non-phosphate builders are required in some locales. Preferred phosphate builders are water-soluble alkali-metal salts of phosphates, pyrophosphates, orthophosphates, tripolyphosphates, higher polyphosphates, and mixtures thereof.

Examples of silicate builders are the alkali metal silicates, particularly those having a SiO₂:Na₂O ratio in the range 1.6:1 to 3.2:1 and layered silicates, such as the layered sodium silicates described in U.S. Patent 4,664,839, issued May 12, 1987 to Rieck. NaSKS-6 is the trademark for a crystalline layered silicate marketed by Hoechst (commonly abbreviated herein as "SKS-6"). Unlike zeolite builders, the Na SKS-6 silicate builder does not contain aluminum.

Examples of carbonate builders are the alkaline earth and alkali metal carbonates as disclosed in German Patent Application No. 2,321,001 to Jacobsen, et al., published November 15, 1973.

Aluminosilicate builders are useful in the present invention. Aluminosilicate builders include those having the empirical formula:

15 $M_z(z\text{AlO}_2)_y \cdot x\text{H}_2\text{O}$
wherein z and y are integers of at least 6, the molar ratio of z to y is in the range from 1.0 to about 0.5, and x is an integer from about 15 to about 264.

Organic detergent builders suitable for the purposes of the present invention include, but are not restricted to, a wide variety of polycarboxylate compounds. As used herein, "polycarboxylate" refers to compounds having a plurality of carboxylate groups, preferably at least 3 carboxylates. Polycarboxylate builder can generally be added to the composition in acid form, but can also be added in the form of a neutralized salt. When utilized in salt form, alkali metals, such as sodium, potassium, and lithium, or alkanolammonium salts are preferred.

Citrate builders, e.g., citric acid and soluble salts thereof (particularly sodium salt), are polycarboxylate builders of particular importance for heavy duty liquid detergent formulations due to their availability from renewable resources and their biodegradability. Citrates can also be used, especially in combination with zeolite and/or layered silicate builders. Oxydisuccinates are also especially useful in such compositions and combinations.

3. Bleaching Compounds - Bleaching Agents and Bleach Activators

The detergent compositions herein may further contain bleaching agents or bleaching compositions containing a bleaching agent and one or more bleach activators. When present, bleaching agents will typically be at levels of from

about 1% to about 30%, more typically from about 5% to about 20%, of the detergent composition, especially for fabric laundering. If present, the amount of bleach activators will typically be from about 0.1% to about 60%, more typically from about 0.5% to about 40% of the bleaching composition comprising the 5 bleaching agent-plus-bleach activator. For a more detailed description and preferred bleaching compounds, including oxygen bleach and bleach activators, please see the description above.

4. Brightener

Any optical brighteners or other brightening or whitening agents known in 10 the art can be incorporated at levels typically from about 0.05% to about 1.2%, by weight, into the detergent compositions herein. Commercial optical brighteners which may be useful in the present invention can be classified into subgroups, which include, but are not necessarily limited to, derivatives of stilbene, pyrazoline, coumarin, carboxylic acid, methinecyanines, 15 dibenzothiphene-5,5-dioxide, azoles, 5- and 6-membered-ring heterocycles, and other miscellaneous agents. Examples of such brighteners are disclosed in "The Production and Application of Fluorescent Brightening Agents", M. Zahradnik, Published by John Wiley & Sons, New York (1982).

Specific examples of optical brighteners which are useful in the present 20 compositions are those identified in U.S. Patent 4,790,856, issued to Wixon on December 13, 1988. These brighteners include the PHORWHITE series of brighteners from Verona. Other brighteners disclosed in this reference include: Tinopal UNPA, Tinopal CBS and Tinopal 5BM; available from Ciba-Geigy; Artic White CC and Artic White CWD, available from Hilton-Davis, located in Italy; the 25 2-(4-styryl-phenyl)-2H-naphthol[1,2-d]triazoles; 4,4'-bis-(1,2,3-triazol-2-yl)-stilbenes; 4,4'-bis(styryl)bisphenyls; and the aminocoumarins. Specific examples of these brighteners include 4-methyl-7-diethyl- amino coumarin; 1,2-bis(-venzimidazol-2-yl)ethylene; 1,3-diphenyl-phrazolines; 2,5-bis(benzoxazol-2-yl)thiophene; 2-styryl-naphth-[1,2-d]oxazole; and 2-(stilbene-4-yl)-2H-naphtho-[1,2-30 d]triazole. See also U.S. Patent 3,646,015, issued February 29, 1972 to Hamilton. Anionic brighteners are preferred herein.

5. Chelating Agents

The detergent compositions herein may also contain one or more iron 35 and/or manganese chelating agents. Such chelating agents can be selected from the group consisting of amino carboxylates, amino phosphonates,

polyfunctionally-substituted aromatic chelating agents and mixtures therein, all as hereinafter defined. Without intending to be bound by theory, it is believed that the benefit of these materials is due in part to their exceptional ability to remove iron and manganese ions from washing solutions by formation of soluble chelates.

Amino carboxylates useful as optional chelating agents include ethylenediaminetetracetates, N-hydroxyethylethylenediaminetriacetates, nitrilotriacetates, ethylenediamine tetrapropionate, triethylenetetraaminehexacetates, diethylenetriaminepentaacetates, and ethanoldiglycines, alkali metal, ammonium, and substituted ammonium salts therein and mixtures therein.

Amino phosphonates are also suitable for use as chelating agents in the compositions of the invention when at least low levels of total phosphorus are permitted in detergent compositions, and include ethylenediaminetetrakis (methylenephosphonates) as DEQUEST. Preferred, these amino phosphonates to not contain alkyl or alkenyl groups with more than about 6 carbon atoms.

Polyfunctionally-substituted aromatic chelating agents are also useful in the compositions herein. See U.S. Patent 3,812,044, issued May 21, 1974, to Connor et al. Preferred compounds of this type in acid form are dihydroxydisulfobenzenes such as 1,2-dihydroxy-3,5-disulfobenzene.

A preferred biodegradable chelator for use herein is ethylenediamine disuccinate ("EDDS"), especially the [S,S] isomer as described in U.S. Patent 4,704,233, November 3, 1987, to Hartman and Perkins.

The compositions herein may also contain water-soluble methyl glycine diacetic acid (MGDA) salts (or acid form) as a chelant or co-builder useful with, for example, insoluble builders such as zeolites, layered silicates and the like.

If utilized, these chelating agents will generally comprise from about 0.1% to about 15% by weight of the detergent compositions herein. More preferably, if utilized, the chelating agents will comprise from about 0.1% to about 3.0% by weight of such compositions.

30 6. Dye Transfer Inhibiting Agents

The compositions of the present invention may also include one or more materials effective for inhibiting the transfer of dyes from one fabric to another during the cleaning process. If used, these agents typically comprise from about 0.01% to about 10% by weight of the composition, preferably from about 0.01% to about 5%, and more preferably from about 0.05% to about 2%. Generally,

such dye transfer inhibiting agents include polyvinyl pyrrolidone polymers, polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, manganese phthalocyanine, peroxidases, and mixtures thereof.

The most preferred polyamine N-oxide useful as dye transfer inhibiting polymers in the detergent compositions herein is poly(4-vinylpyridine-N-oxide) which as an average molecular weight of about 50,000 and an amine to amine N-oxide ratio of about 1:4.

Copolymers of N-vinylpyrrolidone and N-vinylimidazole polymers (referred to as a class as "PVPVI") are also suitable for use herein. Preferably the PVPVI has an average molecular weight range from 5,000 to 1,000,000, more preferably from 5,000 to 200,000, and most preferably from 10,000 to 20,000. (The average molecular weight range is determined by light scattering as described in Barth, et al., *Chemical Analysis*, Vol 113. "Modern Methods of Polymer Characterization".) The PVPVI copolymers typically have a molar ratio of N-vinylimidazole to N-vinylpyrrolidone from 1:1 to 0.2:1, more preferably from 0.8:1 to 0.3:1, most preferably from 0.6:1 to 0.4:1. These copolymers can be either linear or branched.

The present invention also may employ as a dye transfer inhibitor a polyvinylpyrrolidone ("PVP") having an average molecular weight of from about 5,000 to about 400,000, preferably from about 5,000 to about 200,000, and more preferably from about 5,000 to about 50,000.

7. Enzymes

The detergent compositions herein may further contain enzymes. Enzymes are normally incorporated into detergent or detergent additive compositions at levels sufficient to provide a "cleaning-effective amount". The term "cleaning effective amount" refers to any amount capable of producing a cleaning, stain removal, soil removal, whitening, deodorizing, or freshness improving effect on substrates such as fabrics, dishware and the like. In practical terms for current commercial preparations, typical amounts are up to about 5 mg by weight, more typically 0.01 mg to 3 mg, of active enzyme per gram of the detergent composition. Stated otherwise, the compositions herein will typically comprise from 0.001% to 5%, preferably 0.01%-1% by weight of a commercial enzyme preparation. Protease enzymes are usually present in such commercial preparations at levels sufficient to provide from 0.005 to 0.1 Anson units (AU) of activity per gram of composition. For certain detergents, such as in automatic

dishwashing, it may be desirable to increase the active enzyme content of the commercial preparation in order to minimize the total amount of non-catalytically active materials and thereby improve spotting/filming or other end-results. Higher active levels may also be desirable in highly concentrated detergent 5 formulations. For a more detailed description and preferred enzymes, please see the description above. For a more detailed description, please see the description above.

8. Polymeric Soil Release Agent

Known polymeric soil release agents, hereinafter "SRA", can optionally be 10 employed in the present detergent compositions. If utilized, SRA's will generally comprise from 0.01% to 10.0%, typically from 0.1% to 5%, preferably from 0.2% to 3.0% by weight, of the compositions. For a more detailed description and preferred SRA's, please see the description above.

9. Suds Suppressors

The compositions herein may contain from 0% to about 5% of suds suppressor. When utilized as suds suppressors, monocarboxylic fatty acids, and salts therein, will be present typically in amounts up to about 5%, by weight, of the detergent composition. Preferably, from about 0.5% to about 3% of fatty 15 monocarboxylate suds suppressor is utilized. Silicone suds suppressors are typically utilized in amounts up to about 2.0%, by weight, of the detergent composition, although higher amounts may be used. This upper limit is practical in nature, due primarily to concern with keeping costs minimized and effectiveness of lower amounts for effectively controlling sudsing. Preferably from about 0.01% to about 1% of silicone suds suppressor is used, more 20 preferably from about 0.25% to about 0.5%. As used herein, these weight percentage values include any silica that may be utilized in combination with polyorganosiloxane, as well as any adjunct materials that may be utilized. Monostearyl phosphate suds suppressors are generally utilized in amounts ranging from about 0.1% to about 2%, by weight, of the composition. 25 Hydrocarbon suds suppressors are typically utilized in amounts ranging from about 0.01% to about 5.0%, although higher levels can be used. The alcohol suds suppressors are typically used at 0.2%-3% by weight of the finished compositions. For a more detailed description, please see the description above.

D. Laundry Methods

In a manual laundry method, the method typically comprises contacting and/or treating soiled fabric with an aqueous wash solution in a bucket or a container with a solid bar. The consumer contacts the solid bar with the soiled fabric by scrubbing. After all the fabric has been scrubbed, fresh water is added 5 to the container and the fabrics are rinsed. This rinsing process may be repeated. During a typical manual laundry method, a cleaning or scrubbing implement may also be used.

EXAMPLES

The following examples further describe and demonstrate embodiments 10 within the scope of the present invention. The examples are given solely for the purpose of illustration and are not to be construed as limitations of the present invention, as many variations thereof are possible without departing from the spirit and scope of the invention.

In the following Examples all levels are quoted as % by weight of the 15 composition. The following examples are illustrative of the present invention, but are not meant to limit or otherwise define its scope. All parts, percentages and ratios used herein are expressed as percent weight unless otherwise specified.

Examples 1-5

The following are example encapsulated detergent particles compositions 20 in accordance with the present invention:

By weight of the encapsulated detergent particle

	1	2	3	4	5
Detergent active 1	90				
Detergent active 2		80			
25 Detergent active 3			70		
Detergent active 4				70	
Detergent active 5					60
Coating material 1	10		30		40
Coating material 2		20		30	

30 Detergent active 1 - perborate monohydrate

Detergent active 2 - sodium percarbonate

Detergent active 3 - acrylic-maleic co-polymer

Detergent active 4- ethoxylated polyethyleneimine

35 Detergent active 5 - di-tallow di-methyl ammonium chloride

Coating material 1- N1-hydroxyethyl, N2,N3,dimethyl,N3-dodecyl diethylenetriamine

Coating material 2 - Schiff base compound

The above encapsulated detergent particles are made by the following process: Take 500 grams of a detergent active such as perborate monohydrate. Spray 20% of a coating material, such as coating material 1, solution at room temperature, or at 20-50°F above the melting point of the amine coating. Monitor the weight of the coating solution applied. After about 125 grams of the coating solution is applied, the coating process is completed. The amount of coating applied is approximately 20% by weight of the encapsulated detergent particle.

Example 6

The encapsulated particle of Example 1 was formulated with other conventional detergent ingredients and formed into a laundry detergent bar composition. The detergent active is stable during the bar process as well as during storage, and the final bar composition has good cleaning ability.

It is understood that the examples and embodiments described herein are for illustrative purposes only and that various modifications or changes in light thereof will be suggested to one skilled in the art without departing from its spirit and scope.

WHAT IS CLAIMED IS:

1. An encapsulated detergent particle comprising a detergent active encapsulated with a coating material enabling a delayed release of the detergent active into the wash solution, the coating material being insoluble in a wash solution having a pH of equal to or greater than 10 at 25°C and such coating material being soluble in a wash solution having a pH of equal to or less than 9, wherein the coating material has a protonation constant of from 10 to 8 and is selected from the group consisting of amines, Schiff base compounds, and mixtures thereof..
5
2. The encapsulated detergent particle according to Claim 1, wherein the detergent active is selected from the group consisting of oxygen bleaches, bleach activators, polymeric soil release agents, enzymes, fabric softening agents, bactericides, bacteriostats, suds suppressors, and mixtures thereof.
3. A detergent bar composition comprising:
 - (a) from about 10% to about 60% by weight of a deterutive surfactant;
 - (b) from about 5% to about 60% by weight of a detergent builder; and
 - (c) an encapsulated detergent particle comprising a detergent active encapsulated with a coating material enabling the delayed release of the detergent active into the wash solution, the coating material being insoluble in a wash solution having a pH of equal to or greater than 10 at 25°C and such coating material being soluble in a wash solution having a pH of equal to or less than 9; wherein the detergent active is selected from the group consisting of oxygen bleaches, bleach activators, polymeric soil release agents, enzymes, optical brighteners, dye transfer inhibiting agents, and mixtures thereof; and wherein the coating material has a protonation constant of from 10 to 8 and is selected from the group consisting of amines, waxes, Schiff base compounds, and mixtures thereof.
5
 - 10
4. The bar composition according to Claim 3, wherein the coating material is selected from the group consisting of amines, Schiff base compounds, and mixtures thereof.
15

5. The bar composition according to Claim 3, wherein the detergent composition is a solid bar composition, wherein the bar is formed from an extruded, shear mixed dough.
6. The bar composition according to Claim 3, wherein the detergent builder is a phosphate builder selected from the group consisting of water-soluble alkali-metal salts of phosphates, pyrophosphates, orthophosphates, tripolyphosphates, higher polyphosphates, and mixtures thereof.
7. The bar composition according to Claim 3, wherein the detergent active is a peroxygen bleach selected from the group consisting of alkali metal salts of perborate, percarbonate, and mixtures thereof.
8. A detergent bar composition comprising:
 - (a) from about 10% to about 60% by weight of a deterotive surfactant selected from the group consisting essentially of C10-18 linear alkyl benzene sulfonates, C10-18 alkyl sulfates, and mixtures thereof;
 - (b) from about 5% to about 60% by weight of a phosphate detergent builder selected from the group consisting essentially of water-soluble alkali-metal salts of phosphates, pyrophosphates, orthophosphates, tripolyposphates, higher polyphosphates, and mixtures thereof; and
 - (c) an encapsulated detergent particle comprising a detergent active encapsulated with a coating material enabling the delayed release of the detergent active into the wash solution, the coating material being insoluble in a wash solution having a pH of equal to or greater than 10 at 25°C and such coating material being soluble in a wash solution having a pH of equal to or less than 9; wherein the detergent active is selected from the group consisting of oxygen bleaches, bleach activators, polymeric soil release agents, enzymes, optical brighteners, dye transfer inhibiting agents, and mixtures thereof; and wherein the coating material has a protonation constant of from 10 to 8 and is selected from the group consisting of amines, waxes, Schiff base compounds, and mixtures thereof.
10. The bar composition of Claim 8, wherein the detergent active is an oxygen bleach selected from the group consisting of perborate, percarbonate, and mixtures thereof.
15. A washing method for cleaning soiled fabric comprising contacting the fabric with a wash solution comprising the bar composition according to Claim 3 and water,

such that the wash solution has a pH of equal to or greater than 10 at 25°C during
the wash, and subsequently contacting the fabric with a rinse solution having a pH
of equal to or less than 9, thereby enabling the delayed release of the detergent
active.

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/US 98/19917

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C11D17/00 C11D3/30 C11D3/26 C11D3/18 C11D1/37
C11D3/065 //C11D3/39,C11D3/37,C11D3/386,C11D1/62,C11D3/48,
C11D3/42,C11D1:14,C11D1:22

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 221 220 A (UNILEVER) 31 January 1990 31 January 1990 see abstract see claims see examples	1-3,5-10
A	US 4 728 453 A (CHOY CLEMENT K) 1 March 1988 see claims 1,5,7-10 see examples	1,2
A	EP 0 397 246 A (PROCTER & GAMBLE) 14 November 1990 see claims 1,3,4,7-9,12,14-16 see page 2, line 12 - line 15 see examples	1,3-5,8

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

Date of mailing of the international search report

27 May 1999

04/06/1999

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INTERNATIONAL SEARCH REPORTInte onal Application No
PCT/US 98/19917**C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT**

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

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